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I also certify that by virtue of an assignment registered under the Patents Act 1977, the application is now proceeding in the name as substituted.

I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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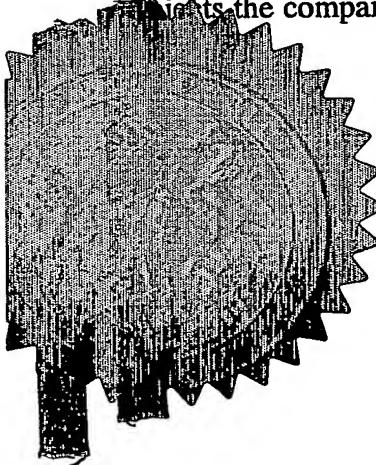
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Dated 28 April 2003

*Stephen Hordley*





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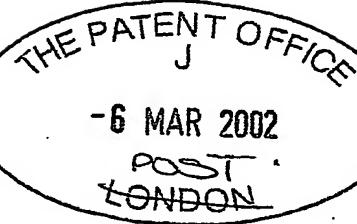
GB0205543.2

By virtue of a direction given under Section 30 of the Patents Act 1977, the application is  
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BACCHUS TECHNOLOGIES LTD  
Incorporated in the United Kingdom  
The Cider Mill, Fair Oaks Farm  
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ADP No. 08572836002

PATENTS ACT 1977



- 6 MAR 2002

PATENTS FORM No 1/77

The Comptroller,  
The Patent Office

11MAR02 E702112-2 D02626  
P01/7700 0.00-0205543.2

## REQUEST FOR GRANT OF A PATENT

THE GRANT OF A PATENT IS REQUESTED BY THE UNDERSIGNED ON THE  
BASIS OF THE PRESENT APPLICATION

I Agent's reference: TAYLOR

0205543.2

II Title of Invention: Corks and Closures

III Applicant:

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SECTION 30 (1977 ACT) APPLICATION FILED 17.02.03  
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VII Declaration of Priority:

Nil

FS1/27 7429210007  
"1/4/03

VIII The Application claims an earlier date under Section 8(3), 12(6), 15(4), or 37(4):  
Nil

IX Check List

A The application contains the following numbers of sheets:

1 Request 1 sheet

2 Description 8 sheets

3 Claims nil

4 Drawings nil

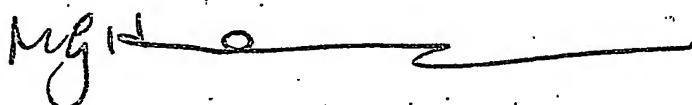
5 Abstract nil

B The application as filed is accompanied by:

nil

X It is suggested that Figure No. of the drawings should accompany the abstract when published

XI Signature:



For the Applicants

## Corks and Closures

5 The present invention relates to corks and similar closures.

Corks have traditionally been used to close wine bottles (both still and sparkling), spirit bottles, and the like. The traditional material is natural cork, which is obtained from the bark of certain oak trees. The cork is inserted into the  
10 neck of the bottle and expands due to its inherent elasticity to seal the neck of the bottle, preventing air from ingressing and wine from escaping.

More recently, various modern variants have been introduced. In some cases, these use synthetic plastics materials; in others, they use natural cork which  
15 has been reconstituted in some way, alone or in combination with synthetic materials. Examples of synthetic closures are described, for example, in WO 96/34806. These are typically cylinders of elastomers such as polypropylene, polyethylene and vinyl acetate. The elastomers are melted and mixed with blowing agents which create a foam. The mixture is then typically either injected  
20 into a mould or extruded and sliced to produce synthetic closures of shapes and sizes which are similar to natural corks.

Synthetic closures can also be made of foamed resins such as polystyrene or mixtures of polystyrene and other chemicals such as butadiene rubber. These  
25 are manufactured by injecting beads of expandable polystyrene into moulds. The beads themselves contain a blowing agent, typically pentane. Steam is injected into the mould and the beads swell up and stick together, taking the cylindrical shape of the mould.

30 A comparative trial of synthetic closures versus natural corks, run by the Australian Wine Research Institute and whose results were published in the

Australian Journal of Grape and Wine Research in 2001, showed that in general synthetic closures do not fully reproduce the sealing capabilities of natural cork. In particular, wine bottled using synthetic closures tends to oxidize faster than wine bottled using quality natural corks. This is partly due to the penetration of 5 oxygen through the interface between the synthetic material and the glass bottle, but also, we have surprisingly found, through the body of the synthetic material. Whilst the rate of oxygen permeation is small, over time the amount of oxygen getting through can be significant.

10 Our research has found that the polymer chains which make up the elastomers and resins are large molecules which surprisingly provide space for small oxygen molecule to diffuse through. The core of a synthetic closure is foamed to provide much of the elasticity of the closure, so the amount of polymer through which a molecule of oxygen has to pass is small relative to the length of 15 the closure. Different polymers have different rates of permeability to oxygen. The permeability rates for the following polymers commonly found in synthetic closures are as follows:

Polystyrene	933
Polypropylene	1952
Polythene	4669

20 all in cc/sq m/ day for a 1mm thickness. Whilst polystyrene has a better, ie lower, rate of permeability, it tends to suffer from microscopic pores between the fused beads of polystyrene. This allows significantly more gas to pass through the closure than would pass through an extruded or injection moulded closure where 25 no such pores exist.

There have been concerns about the possible permeability of both natural corks and synthetic closures. With natural corks, a wax plug has been used as an additional barrier on the top end of the cork; this is in fact provided primarily for 30 aesthetic effect. With synthetic closures, WO 96/34806 (Betacorque) has

proposed the use of a flexible impervious coating such as polyurethane on the ends of the closure if an additional degree of impermeability is required.

5 The main object of the present invention is to provide a closure member which provides improved control of gas penetration, particularly oxygen penetration, preferably to a low level.

10 There are a number of materials which are high oxygen barrier materials. However, in general these materials have properties (such as too low an elasticity, brittleness, bonding problems, or inadequate as the primary material of synthetic closures) which render them unsatisfactory for present purposes. Many high barrier materials are mainly highly crystalline and hence non-elastic; others are stretchable but with little or no recovery.

15 Some polymers exist which have relatively high gas-barrier capabilities, although they cannot be classified as high gas barriers. Their use for present purposes would therefore preferably require a relatively thick layer. Some of these polymers are based on elastomers such as polypropylene and polyethylene. These incorporate other large molecules within the polymers such as butyl rubber 20 in an attempt to eliminate the spaces between the polymer chains through which gases can pass. This greatly reduces the passage of oxygen, although it does not eliminate it completely. The incorporation of these additional molecules within the polymers tends to reduce their elasticity, which makes them unsuitable for use as the main constituent of synthetic closures. An example of such a material is 25 Oxylon CS25, which is produced by DS Chemie. The Mocon oxygen transmission rate for Oxylon CS25 is 500cc through a 100 micron sheet per square metre per day. Such materials are flexible and have a degree of elastic ability but wrinkle when compressed. Similarly, reactive hot melt polyolefines (RHMPs) are relatively high barrier materials but are relatively rigid when set. Other high 30 barrier materials include ethyl vinyl alcohol (EVOH), polyvinylidene chloride

(PVDC), and PTFE. These have barrier properties of between 3 and 6 cc/s m/day for a 2.5 micron thickness.

There are also non-polymeric high barrier materials such as metal foils, 5 vacuum deposited metals, metal oxides and other oxides such as silicon oxide. The latter three are vacuum deposited onto substrates such as polythene to make gas proof bags for food preservation. These have barrier properties of typically 0.5 cc/sq m/day for a 12 micron deposit of aluminium. However, these have bonding problems and, although they are elastic, have poor resilience.

10

In summary, no coatings are currently known to exist which provide high levels of gas impermeability combined with elasticity, good adhesion, and the capability of being used in contact with food.

15

According to its main feature the present invention provides a closure member having a low permeability barrier extending across its length, wherein the barrier includes a layer of hot melt (HM) polyolefine adhesive.

20

The hot melt (HM) polyolefine is preferably a reactive hot melt polyurethane. Reactive Hot Melt Polyurethanes (RHMPs) are derived from the mixing of two components: an isocyanate rich solution and a polyol-rich solution. (This is in contrast to ordinary polyurethane coatings, which consist of a polyurethane dissolved in a solvent.) The polyol is mostly polyester and/or acrylic polyols. There are two groups of polyurethanes - aromatic (based on aromatic isocyanates and mostly polyether polyols) and aliphatic (based on aliphatic isocyanates and /or polyester and acrylic polyols). Aliphatic polyurethanes are preferred, since aromatic polyurethanes have a potential health hazard.

25

With an RHMP, a rapid exothermic chemical polymerisation reaction occurs when the two components are mixed, resulting in a polyurethane pre-

polymer. This pre-polymer is what is applied to the closure. It is applied by being melted and spread. The reaction is irreversible under the influence of heat, preventing it from returning to its pre-melted state after use and improving its barrier properties. Further reactions after application occur under the influence of moisture, resulting in a high level of cross linkage between polymers. The resulting polymer is very stable and has a low level of free monomers which could otherwise taint wine.

RHMPs bond very well to polymer based synthetic closures as well as to natural corks. RHMPs are flexible but relatively inelastic when set. However, we have found that when they are stretched or compressed, they become much more elastic. This elasticity allows the gas barrier to move with the closure. In addition, we have found that the close bonding between the layer of RHMP and the bulk material of the closure results in the elasticity of the bulk material being partially induced or transferred into the RHMP. As a result, the closure can be used in the same way as ordinary corks or synthetic closures, while the RHMP layer forms a relatively low permeability oxygen barrier.

Further materials such as metal oxides, eg iron oxide, can be added to the RHMP to improve its oxygen barrier properties.

We have also found that there is a further advantage to the use of an RHMP barrier layer. This elasticity of the RHMP layer provides the force necessary to achieve the seal required; in addition, where the RHMP layer extends to the edge of the closure, it makes contact with the glass surface and acts as a gasket preventing gas ingressing and wine exiting along the closure/glass interface. Since it is a substantially denser and tougher material than that of the main synthetic material of the closure, it exerts a considerable extra force on the glass over a small area, so providing a strong seal.

In known closures, the barrier is located at an end of the closure member. In the present invention, the barrier may be applied to one or both ends of the closure, but is preferably included in the body of the closure, with the two ends of the closure both being bonded to the barrier to form a unitary closure member.

5

The barrier can be applied anywhere along the length of the body of the closure across the long axis, so that it forms a partial or complete barrier to the movement of gases and fluids. The barrier preferably extends from one side of the closure to the other, thus providing a continuous barrier. However, it can be 10 applied partially, for example only covering the central part and thus providing an improvement to the permeability without providing a complete barrier. It can also be used to provide total coverings. If desired, one or more further barriers may be provided at further positions along the closure.

15

The barrier can also extend slightly beyond the cross sectional area, providing a gasket which sits proud of the surface of the closure. Provided that the barrier is elastic enough, the small area of the gasket exerts a high point load around the circumference of the closure/glass interface. This provides a much improved seal to prevent gases passing up the interface. Most synthetics use 20 silicone lubricants, which are not good oxygen barriers, to ensure smooth insertion and extraction characteristics. (In contrast, natural corks usually use paraffin wax or paraffin wax/silicone mixtures, paraffin wax being a good oxygen barrier.)

25

The gas barrier layer may be thick or thin. A thin layer is preferable as it takes on the elasticity of the body more effectively and does not look odd on extraction due to swelling significantly beyond the surface of the closure. However, having a thick gas barrier is more effective at inhibiting gas movement for some oxygen barrier materials.

30

The above closure, with its RHMP layer, provides a closure with relatively low permeability. According to a further major feature of the invention, however,

there is provided a closure in which the barrier includes a further layer of material having lower permeability than RHMP.

5 This permits the barrier layer to be thin while achieving very low permeability. Typically the thickness of the RHMP can range from 1 micron up to 2000 microns, and the low permeability material can range from 1 micron to 1000 microns. Multiple layers of RHMP and barrier materials may be applied.

10 The low permeability layer can conveniently be attached to the main body of the closure by the RHMP, and if the barrier is not at the end of the closure, the low permeability layer can conveniently be attached to both parts of the main body by RHMP layers on both sides.

15 We have found however that certain types of low permeability material can be formed as a layer directly on the bulk material of the closure. Thus in some cases the barrier can be fixed to the closure by any suitable means such as fusion by heat, spraying in a suitable solvent, incorporation in the mould, physical bonding, chemical bonding, vapour deposition, or melting and application by printing or rolling on. A barrier which is applied as a liquid can be applied by any 20 means such as direct application, brushing, printing, spraying, or dipping. In all such cases, however, an RHMP layer is also used. If the barrier is not at the end of the closure, the RHMP secures the open side of the low permeability layer to the second half of the closure; if the barrier is at the end of the closure, the RHMP layer is applied to its outer surface.

25

Where there is an RHMP layer between the low permeability layer and the substrate, it ensures good adhesion between the low permeability layer and the substrate. In addition to this direct effect, the RHMP layer aids in effectively transferring or inducing the elastic properties of the substrate into the low 30 permeability layer. The more powerful elastic response of the RHMP, combined with its strong adhesive capabilities, forces the low permeability layer to conform

with the movements of the RHMP layer. The chance of fracture in a low permeability layer of brittle material is thus greatly reduced. (Further, if such fracture should occur, its extent is likely to be greatly reduced, and its effects minimized as the RHMP will keep the pieces in place.)

5

The present invention may be contrasted with the closures of WO 96/34806 patent mentioned above. That described only polyurethane resins as sealants, not hot melt (HM) polyolefines or reactive hot melt polyurethanes (RHMPs). At that time, the movement of oxygen through materials was not understood and the 10 resins were designed only to stop the gross movement of gas and moisture.

15

The present system can be applied to natural corks as well as synthetic closures to improve their gas permeability. In the case of natural corks, the present barrier will also act as a barrier to microbiological contaminants such as cork-taint (eg trichloroanisole (TCA)) and yeasts which contaminate wine, as well as acting as a barrier to chemicals in the cork which could otherwise enter the wine such as tannins and tars.

20

25

### Claims

Any feature of novelty or combination thereof within the meaning of 30 Article 4H of the International Convention (Paris Convention).



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